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Vacuum Condensing Point 와 물질이동에 관한 Sublimatography (III)

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Mass Transfer and Vacuum Condensing Point of Sublimatography (Ⅲ).

—Relations among the Characters of Sulfur in the Sublimato-tube—

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Abstract

We studied on the sublimatographic characters of sulfur as follows;

1) Vacuum condensing point of sulfur at different heating temperature and vacuum degree is obtained.

2) Amount of sublimation of sulfur is determined quantitatively by the sublimatography.

3) The vapor pressure, molar velocity of sublimation and mass transfer coefficient of sulfur are calculated by experimental data as follows;

$$P_h/P_s=\gamma \quad (\gamma=1.03)$$
$$P_s^n=k\ G \quad (n=0.977)$$
$$(\gamma P_s)^{n'}=k'G \quad (n'=1.0)$$
$$k_{gav}=1.3985\times10^{-2}$$

Introduction

The literature on the separation of the mixture of organic isomers by sublimatography and the application has been well reviewed elsewhere. [1)~16)] In the previous paper, [17)] we have suggested that the relation between the vapor pressures(P_s, P_h) at vacuum condensing point (V. C. P.), heating temperature(t_h) and the sublimation velocity(G) was linearly coincident and the relations among the molar velocity of sublimation(N), the vapor pressures and mass transfer coefficient(k_g) in the sublimato-tube were

$$N=k_g(\gamma-1)P_s, \quad \gamma=P_h/P_s.$$

As a part of the studied on the mass transfer and the vacuum condensing point of sublimatography, the purpose of this study is the application of sublimatography to the character of sublimation of sulfur that have not been studied yet.

Experimental

Material.—Sulfur is purified by sublimation from C. P. grade chemicals (Katayama chemical Co., LTD.)

Apparatus.—Sublimatoscope is the same as that previously described.[17]

Measurements.—1) About 0.05~0.1g. of sulfur is accurately weighed in the weighing tube (Fig. 1) and put in the sample chamber of sublimato-tube.

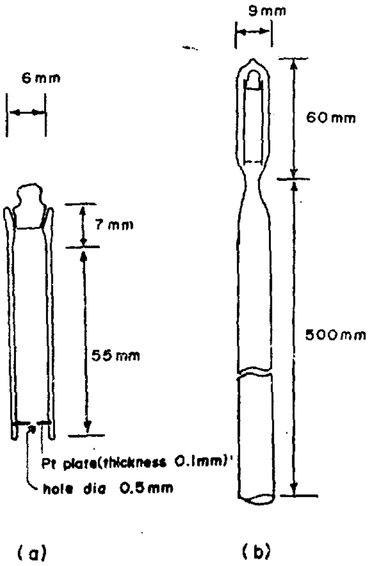

Fig. 1. Weighing tube (a) & Sublimato-tube (b)

2) Sublimato-tube is surrounded by the constant temperature gradient furnace controlled to within ± 1°C and about 20 minutes of standing the tube in the bath is sufficient to obtain complete temperature equilibrium.

3) All of the heating temperatures and vacuum condensing points of sulfur are measured by a vacuum system consisting of a sublimato-tube, a Mcleod gage and Geissler tube for vacuum degree.

4) After each sublimation, a quantity of sulfur could be measured to an accuracy of 0.1% by micro-balance.

Results & discussion

Relations between vacuum condensing point of sulfur and heating temperature at different vacuum degrees are as Fig. 2. And data of reappearances are in agreement with those expected from t_h-V. C. P. curves of sulfur.

As shown in Fig. 3, the relation between the vacuum degree and vacuum condensing point is as follows;

$$t_c = \alpha + \beta \log P_{va}$$

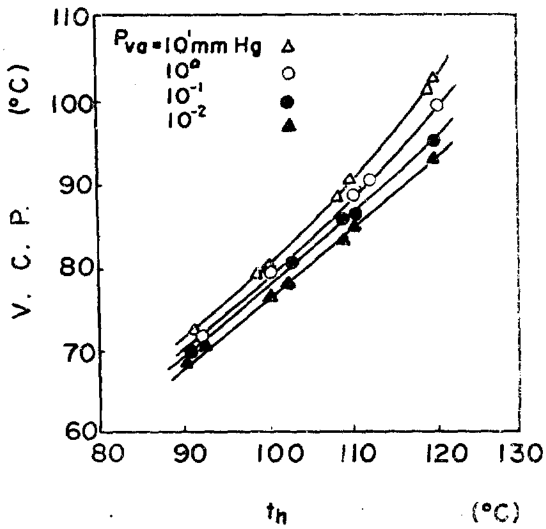

Fig. 2. V. C. P. vs. t_h of sulfur at differerent degree of vacuum

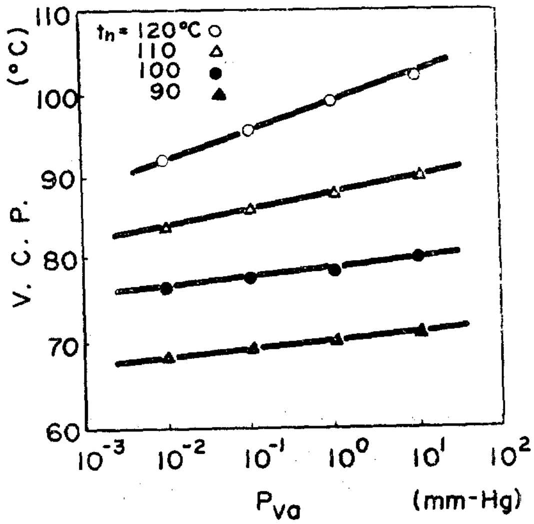

Fig. 3. V. C. P. vs. P_{va} of sulfur of different heating temperature (t_h)

And the quantity of sublimation increased in proportion to heating time. So, a proper heating temperature of sublimatographic separation for sulfur is 110°C($=t_h$) at 10^{-2}mm-Hg of vacuum as shown in Fig. 4.

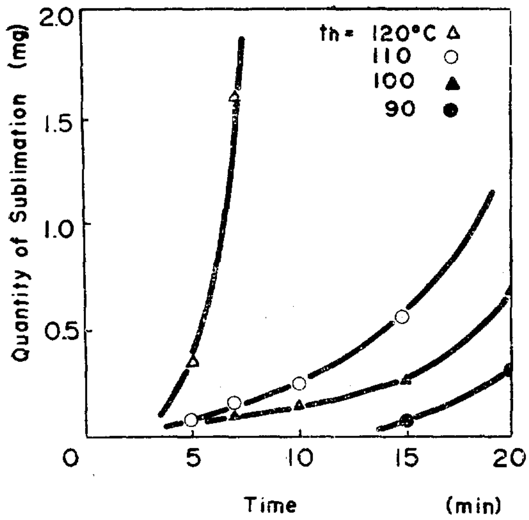

Fig. 4. Quantity of sublimation of sulfur vs. heating time at 10^{-2} mm-Hg, different heating temperature

The results of calculation through Langmir's equation by experimental data are listed in Table 1 and

Table 1. Sublimation Quantity and Saturated Vapor Pressure at t_h-V. C. P. of Sulfur

Exp. No.	1	2	3	4
t_h(°C)	120	110	100	90
V. C. P. (°C)	92	85	76	69
G(g/sec×10^4)	0.1050	0.0367	0.0150	0.0083
N(g·mol/cm²·sec×10^6)	0.8515	0.2974	0.1216	0.0673
γP_s(mm Hg×10^2)	0.1865	0.0643	0.0259	0.0141
P_s (〃)	0.1798	0.0621	0.0251	0.0137
γ	1.0389	1.0342	1.0338	1.0327

γ is 1.03. The mass transfer coefficient in the sublimato-tube are calculated from the equation,

$$k_g=\frac{N}{(\gamma-1)P_s}$$

and are presented in Table 2.

Table 2. k_g of Sulfur

No. \ Eq.	$N=k_g(\gamma-1)P_s$ $\therefore\ k_g=\frac{N}{(\gamma-1)P_s}$
k_{g1}	1.2708×10^{-2}
k_{g2}	1.3962×10^{-2}
k_{g3}	1.4305×10^{-2}
k_{g4}	1.4964×10^{-2}
k_{gav}	1.3985×10^{-2}

The relation between vapor pressure and velocity of sublimation formulated from Fig. 5 and Fig. 6 is expressed by;

$$P_s^n=kG \qquad (n=0.977)$$

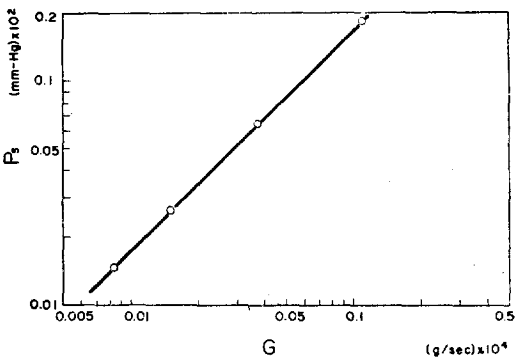

Fig. 5. Plots of P_s vs. G.

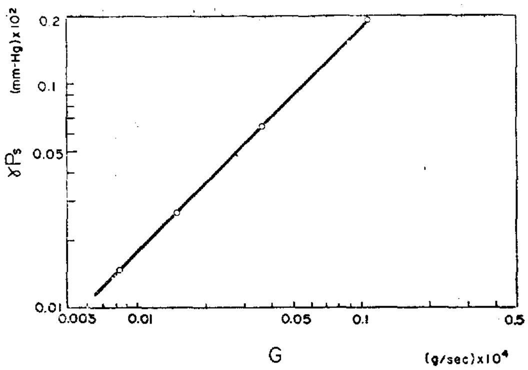

Fig. 6. Plots of γP_s vs. G

$$(\gamma P_s)^{n'}=k'G \qquad (n'=1.0)$$

And Fig. 7 show that we could get the vacuum condensing point by calculation through experimental measurement of sublimation velocity of sulfur.

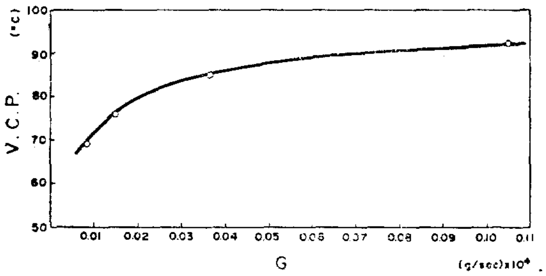

Fig. 7. Plots of V. C. P. vs. *G*

Nomenclatures

t_c: Vacuum condensing point	(°C)
t_h: Heating temperature	(〃)
P_s: Saturated vapor pressure at t_c	(mm-Hg)
P_h: 〃 〃 〃 at t_h	(〃)
γ: P_h/P_s	
N: Molar velocity of sublimation	(g·mol/cm²·sec)
G: Velocity of sublimation	(g·/sec)
k, k': Proportional constant	
α, β: 〃 〃	
k_g: Mass transfer coefficient	(mol/cm²·sec)

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